J. Chem. & Cheml. Sci. Vol.3 (2), 70-78 (2013)

Preparation & Bio-Chemical Identification of Series Organic Compounds

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(Received on: February 12, 2013)

ABSTRACT

This study involved, synthesis of variety of organic compounds [1-6] such as thiol compound, oxazepine(oxazepam), diazepine (diazepam), macrocyclic Schiff base, azo compound which contains electron with donating group and azo compound is containing electron with drawing group and identification of their structures by {(C.H.N)-analysis, H.NMR–spectra, FT.IR– spectra and melting points} and study of their biological activities by biological studies, the data obtained give good supported for synthesized compounds[1-6].

Keywords: Schiff, azo, oxazepine, diazepine, macrocycle.

INTRODUCTION

In this paper, we have used Schiff base condensation as the ring –closing step to synthesize macrocycle[1], oxazepine[2], diazepine[3], thiol compound[4], the heteroatoms in there structure such as (S, N, O) explain variety of applications[(1-4)], antitumor[(5,6)], in the biological engineering[(7)] and in other field[(8-18)] of their specific structures.

Also azo-compounds are synthesized in this research, it is known that aromatic azo compounds are widely used because of azo group (-N=N-)in their structures explain to their activity and variety of applications in several fields [(19-23)].

EXPERIMENTAL

- All chemical used were supplied from Fluka and BDH – Chemical Company
- All measurements were carried out by:

1– Melting points: electro thermal 9300, melting point engineering LTD , U. K.
2– FT.IR-spectra: fourrier transform infrared shimadzu 8300 – (FT.IR), KBr disc was performed.
4 -H-NMR spectra with DMSO-solvent and (C.H.N)-analysis .

Synthesis of compound [1]

The preparation starts with the

reaction between 2,6-di formal-1,4-cresol (0.01mole,1.01 gm) and (0.02mole ,1.2gm) of ethylene diamine for(4hr),the precipitate was filtered off then (0.01mole, 2.48gm) from this precipitate was refluxed with (0.01mole, 1.64gm) of 2,6- diformyl-1,4-cresol for (5 hrs), to precipitate 83% compound [1].

Synthesis of compounds [2-4]:

Refluxing mixture of (0.01mole, 1.36gm) of p-methoxy benzaldehyde with [(0.01mole,1.44gm) of 2-amino quinoline were refluxed for (4hrs), after cooling the precipitate was filtered off and dried, (0.01 mole, 2.62 gm) of this precipitate was condensed with (0.01mole, 0.98gm) of maliec anhydride for (6hr), the precipitate was filtered off to produce 81% of compound[2] ,which (0.01mole, 3.34gm) from it was reacted with one of [(0.01mole,1.23gm) of p-methoxy aniline, (0.01 mole, 1.4gm) of p-methoxy benzene thiol] respectively for (8hrs), after cooling the precipitate was filtered off and recrystallized to produce (80%, 83%) of compounds [3]and[4] respectively.

Synthesis of compound [5]

(0.01mole, 2.21gm) of 3,5-di isopropyl-4-amino phenol was dissolved in 2 ml of hydrochloric acid and (0.7gm) of sodium nitrite in ice medium (0-5)C˚, after that, the ethanolic solution of p-methoxy phenol (0.01mole, 1.24gm) adds to reaction mixture with solution of sodium hydroxide, the precipitate was filtered off and recrystallized to produce (87%) of compounds[5].

Synthesis of compound [6]

Amixture of (0.01mole, 1.25gm) of p-chloro aniline and(0.01mole,0.76gm) of ammonium thiocyanate in glacial acetic acide with bromine addition from burete drop by drop, the precipitate was filtered off and dried, which dissolves (0.01mole, 1.83gm) in 2 ml of hydrochloric acid and (0.7gm) of sodium nitrite in ice medium (0-5)C˚, after that, the ethanolic solution of m-di chloro benzene (0.01mole,1.45gm) added to reaction mixture with solution of sodium hydroxide, the precipitate was filtered off and recrystallized to produce (86%) of compound [6].

RESULTS AND DISCUSSION

All synthesized compounds [1-6] have been characterized by their melting points and spectroscopic methods (FT.IR-spectra, (C.H.N)-analysis H.NMR-spectra) with biological studies.

FT.IR-Spectra

In FT.IR spectra ,the reaction is followed by appearance of: absorption band at (1630)cm^{-1} due to azomethine group[3] (-CH=N-)and band at (3420)cm^{-1} due to hydroxyl group (-OH) [4] of phenol in compound[1].

Appearance of absorption band at (1680)cm^{-1} due to carbonyl group (CO-) lactame[4] (CO-NH), absorption band at (170 0)cm^{-1} due to carbonyl group (CO) of lacton (CO-O) in compound[2], which disappeared and other bands are appear at (1675)cm^{-1} due to[4] carbonyl of lactame (CO-NH) of diazepine in compound[3] and

two bands at (3418,1436)cm^{-1} due to hydroxyl group (-OH) and aryl sulphide (Ar-S) respectively in compound [4].

Appearance of band at (1537)cm^{-1} due to azo group (-N=N-)and band at (3481)cm^{-1} due to hydroxyl group (-OH)in compound[5], where as compound [6] appeared band at (1536)cm^{-1} due to azogroup[20] (-N=N-)and band at (811)cm^{-1} due to (C-S) [3] endo cycle of thiazol, and other data of functional groups show in the following ,Table(1) and figures (4-6).

Appearance of these bands are strong evidence to formation of compounds [1-6].

H.NMR- Spectrum

H.NMR- Spectrum of compounds [1-6] showed : singlet signal at 9.96 for one proton of azomethine group(-CH=N-)[3] ,signal at 10.62 for proton of hydroxyl group(-OH)[4] of phenol, signal at 3.88 for protons of (NCH_2-CH_2-N)[3] in compound[1]. Signal at 9.19 for proton of (O-CH-N) oxazepine cycle[4], signal at 4.58 for protons(C-CH=CH-C) in oxazepine cycle of compound[2].

Signal at 10.16 for proton of (N-CH-N)[3] in diazepine cycle[4] ,signal at 3.38 for protons of (C-CH=CH-C) in diazepine cycle ,doublet of doublet signal at 6.77 for protons of phenyl group in diazepine in compound[3].

Signal at 10.1 for proton of hydroxyl group (-OH) ,signal at 5.2 for protons of (C-CH=CH-C) , signal at 7.2 for protons of phenyl group of thiol in compound [4].

Signal at 10.9 for proton of hydroxyl group (-OH) in phenol, signal at 2.2 for protons of ($C(CH_3)_3$) , signal at 3.4 for protons of methoxy group (-OCH_3) in compound [5].

Signal at 6.5 for protonS of phenyl ring in benzothiazole , signal at 7.2 for protons of phenyl ring on azo group in compound [6].And other peaks shown in the following, figures(1-3).

(C.H.N) – Analysis

(C.H.N) –Analysis ,from compared the calculated data with found data of these compounds, the results were comparable , the data of analysis, M.F, names and melting points are listed in table (2).

Biological Effect of compounds[1-6]

Antimicrobial activities of compounds [1-6] were tested using hole method at concentration (1 10^{-3})M of compounds against two type of bacteria (*Staphylllococcus aureus, Klebsiella phenmonia*) and two type of fungi (*Fusoirium, Aspergillus Nigar)* which incubated at 37 C˚ for 24hrs .

All compounds[1-6] exhibit strong inhibition on growth of the bacteria by inhibition of cell wall synthesis ,disruption of cell membranes interference with protein synthesis or interference with nucleic acid synthesis[18]. While compounds[1-6] have no antifungal activity, the obtained data shown in the following, Table (3).

Table (1) : FT.IR data (cm^{-1}) of compounds [1-6]

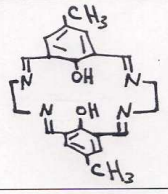
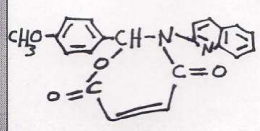
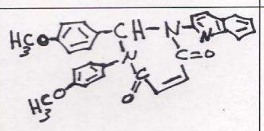
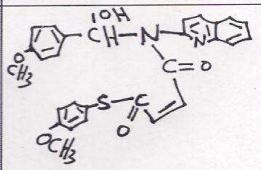
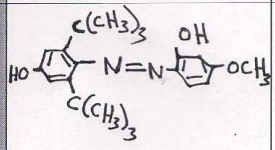
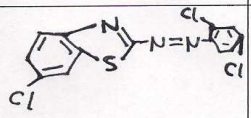
Comp. No.	Structural formula	Name of compound	Functional groups (Importance groups)
[1]		[10,21-dimethyl-3,6,14,17-tetra aza tri cyclo-tetra cosa-2,6,8,10,12,24,13,17,19,23,20,22-decaene-23,24-diol].	(CH=N)azomethine group:1630 (-OH):3420 ,(C-O):1232 (CH)aliphatic:2925 (CH)aromatic:3030
[2]		2-(p-methoxy phenyl)-3-(quinolone)-2,3-dihydro-[1,3]-oxazepine-4,7-dione.	(C=O)of lactame:1680 (C-N)endo cyclic: 1530 (C=O)of lacton:1700 (-OCH_3)2830
[3]		1-(p-methoxy phenol)-2-(p-methoxy phenyl)-3-(quinolone)-1,2,3,-trihydro-[1,3]-diazepine-4,7-dione.	(C=O)of lactame:1675 (C-N)endo cyclic: 1535 (-OCH_3)2820
[4]		N-(p-methoxy benzene alcohol)-4-(p-methoxt phenyl sulphide)-1,4-dion-2-(butane)-N-(2-quinoline) amine.	(C=O)of lactame:1671 (C-N)endo cyclic: 1535 (C=O)of lacton:1700 (-OCH_3)2794 (Ar-S):1436 ,(-OH):3418
[5]		4-(3,5-di iso butyl phenol azo)-anisol.	(-OH):3481 (-OCH_3):2871 (-N=N-):1537
[6]		2-(6-chloro-2-benzothiazolyl azo)-1,5-di chloro benzene.	(C-Cl):729 , (-N=N-):1536 (C-N)endo cycle:1436, (C-S):81

Table (2) : Melting points, M.F , & (C.H.N)- analysis of compounds [1-6]

Comp.No.	M.F	M.P C°	Calc./ Found C%	H %	N %
[1]	$C_{22}H_{24}N_4O_2$	210	70.212 69.948	6.382 6.224	14.893 14.617
[2]	$C_{21}H_{16}N_2O_4$	139	70.000 69.869	4.444 4.288	7.777 7.518
[3]	$C_{28}H_{23}N_3O_4$	159	72.258 72.127	4.946 4.836	9.032 8.879
[4]	$C_{28}H_{24}N_2O_5S$	187	67.200 67.110	4.800 4.520	5.600 5.361
[5]	$C_{21}H_{28}N_2O_3$	174	70.786 70.547	7.865 7.716	7.865 7.667
[6]	$C_{13}H_6N_3SCl_3$	183	45.547 45.326	1.751 1.616	12.262 12.128

Table(3):biological activity of (1 10^{-3})M of compounds[1-6]expressed as zone of Inhibition(mm)

Comp.No.	Bacteria		Fungi	
	S.aureus	K.pheumonia	A.spergillus niger	Fusoirium
[1]	21	16	------	-----
[2]	18	13	------	-----
[3]	23	15	------	------
[4]	25	18	-----	------
[5]	10	9	-------	-------
[6]	14	7	---------	---------

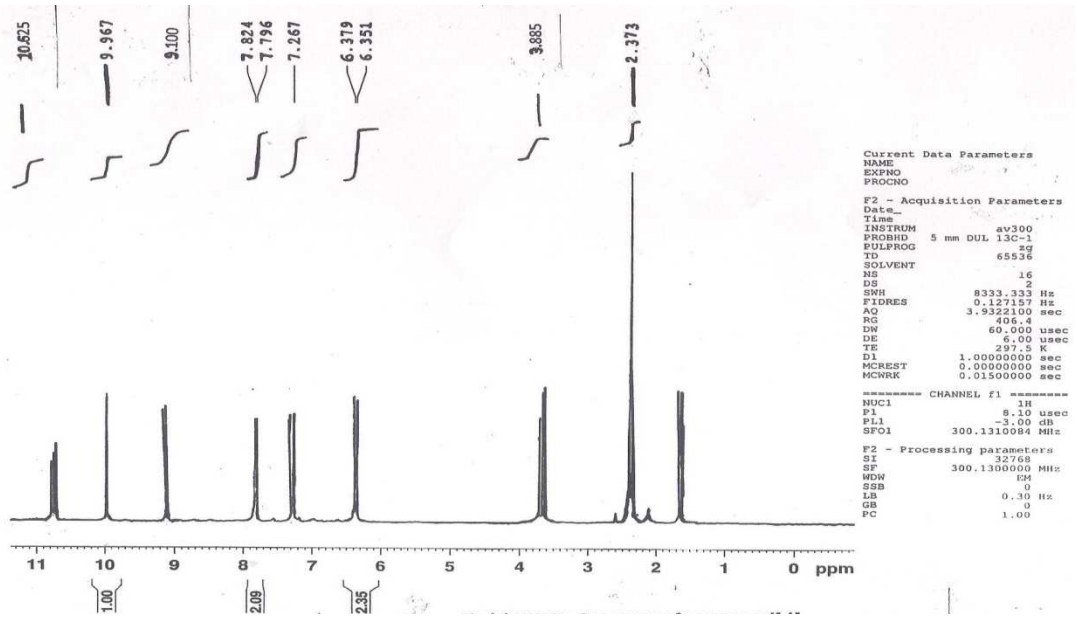


Fig. (1) H.NMR-Spectrum of compound [1]

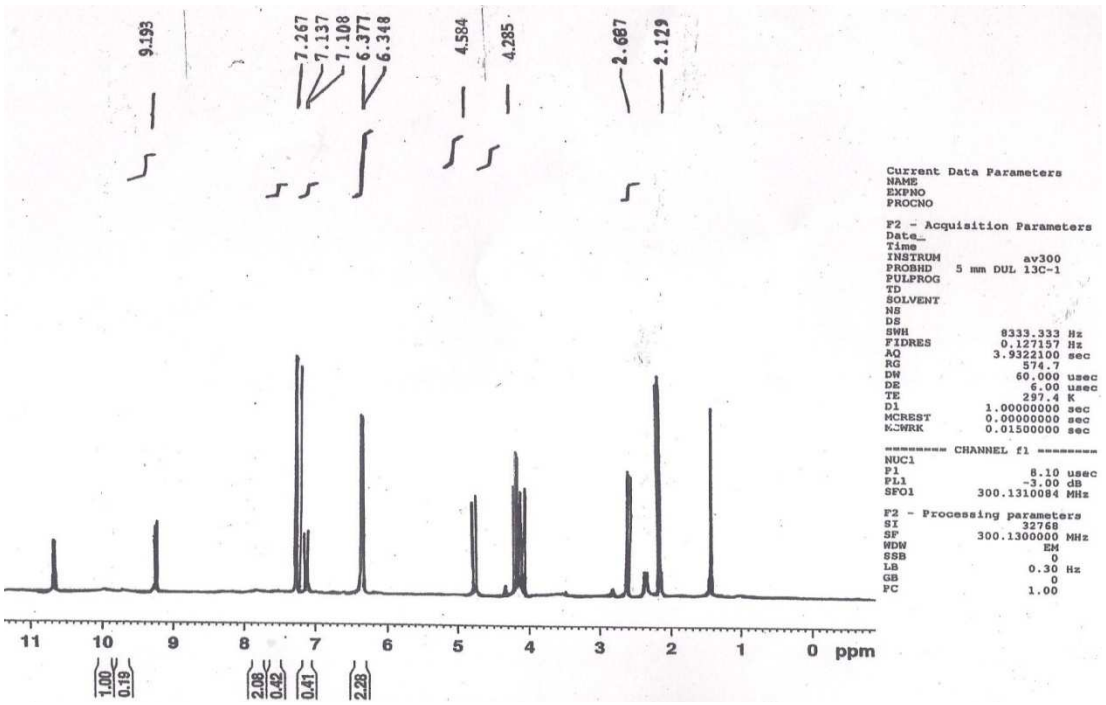


Fig. (2) H.NMR-Spectrum of compound [2]

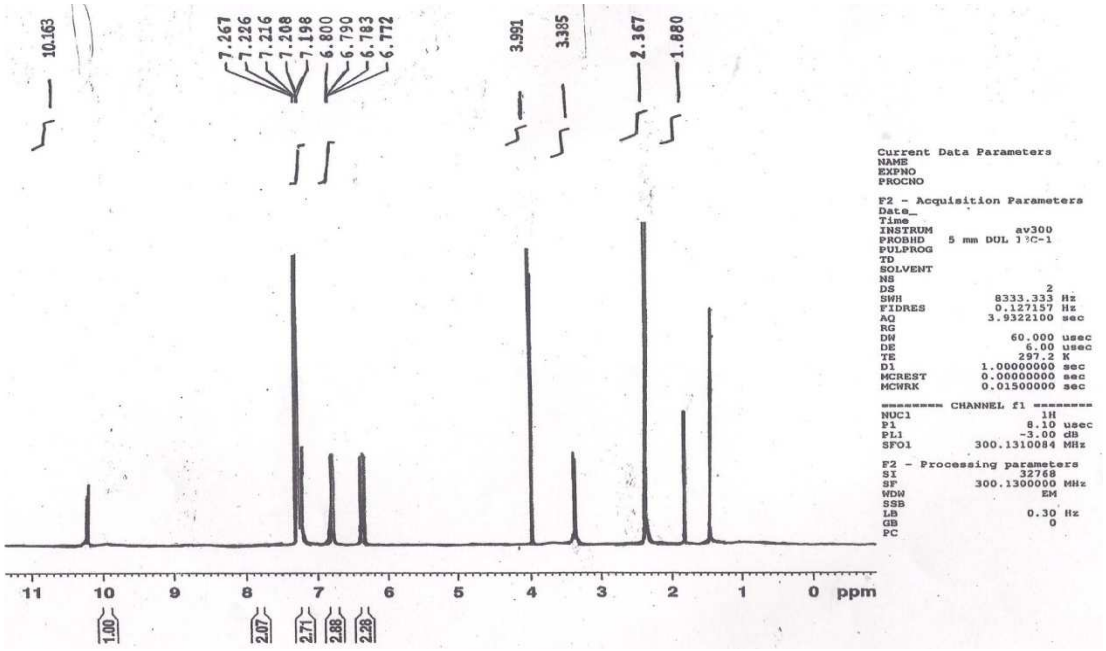


Fig. (3) H.NMR-Spectrum of compound [3]

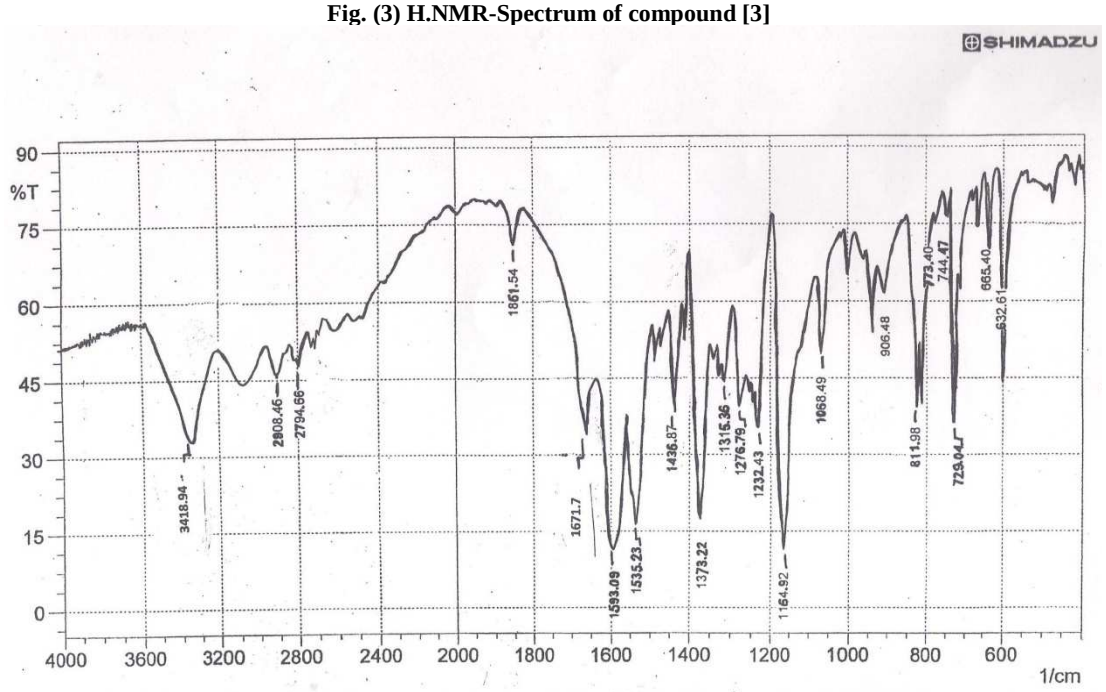


Fig. (4) FT-IR Spectrum of Compound [4]

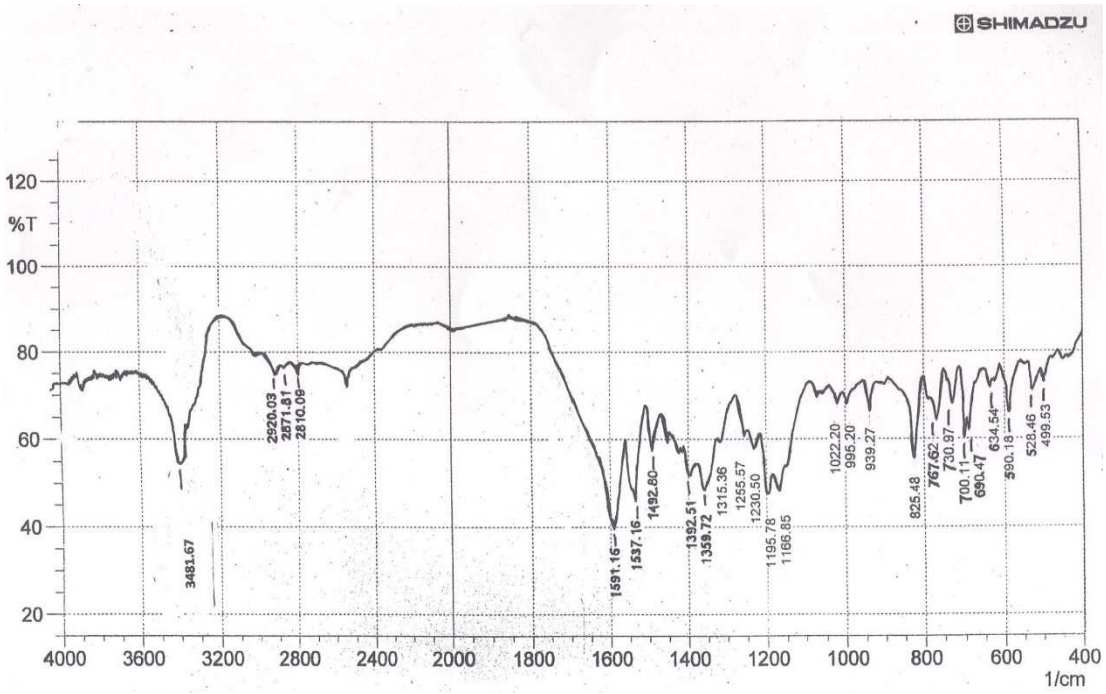


Fig. (5) FT-IR Spectrum of Compound [5]

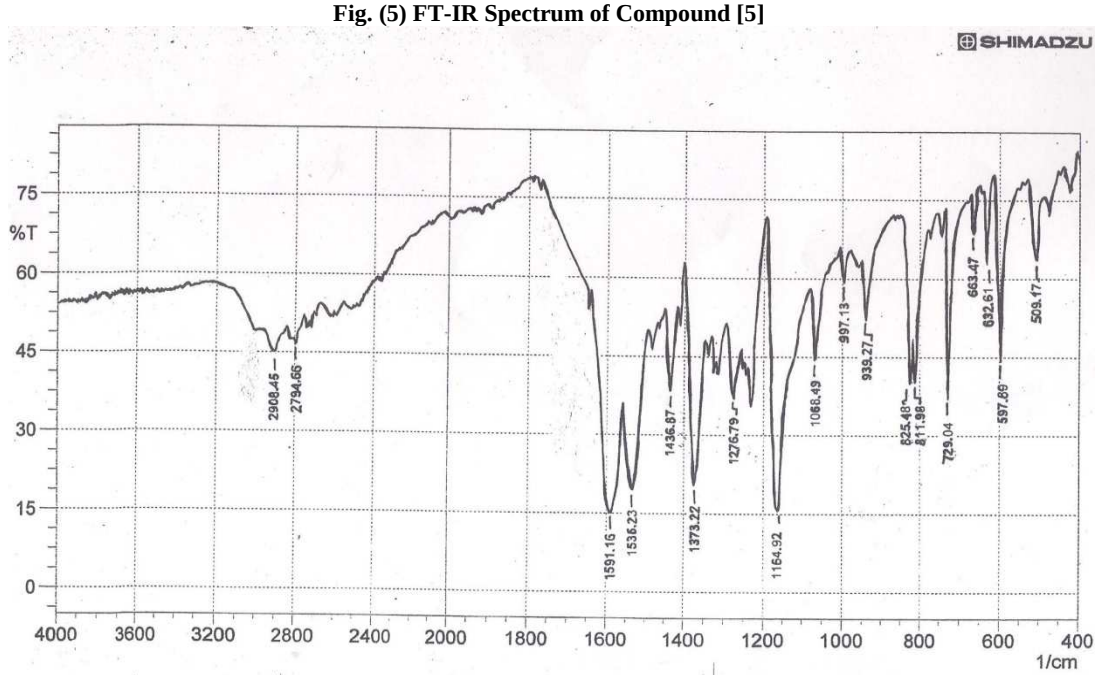


Fig. (6) FT-IR Spectrum of Compound [6]

ACKNOWLEDGEMENT

I would like to express my thanks for Mr. Audai for providing {(C.H.N)-element analytical, H.NMR-spectra and Melting points (digital)} and Zaidan Company for supplied materials.

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